

## Validation of a Method for the Determination of Acetonitrile in Human Exhalate Using Thermal Desorption GC-NPD

Sample Identification	Replicate	Concentration (µg/mL)	Concentration Added (µg/mL)	Percent Recovery (%)	Mean (%)	Standard Deviation (%)	Relative Standard Deviation (%)	Reference Page(s)
Medium QC 30.0µg/mL	1	28.1	30.0	93.7	97.2	3.51	3.61	V6
	2	28.4	30.0	94.7				
High QC 60.0µg/mL	1	60.5	60.0	101	99.2			
	2	59.5	60.0	99.2				

Standards								
0.200µg/mL 6-0432-1G	1	0.201	0.200	101	100	7.9	7.90	V6
0.200µg/mL 6-0432-1G	2	0.213	0.200	107				
1.00µg/mL 6-0432-1F	2	1.13	1.00	113	93.4			
5.00µg/mL 6-0432-1E	1	4.67	5.00	93.4				
20.0µg/mL 6-0432-1D	1	17.9	20.0	89.5	90.0			
20.0µg/mL 6-0432-1D	2	18.0	20.0	90.0				
50.0µg/mL 6-0432-1C	1	52.7	50.0	105	101			
80.0µg/mL 6-0432-1B	1	80.8	80.0	101				
100µg/mL 6-0432-1A	1	101	100	101				

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Sample Identification	Replicate	Concentration (µg/mL)	Concentration Added (µg/mL)	Percent Recovery (%)	Mean (%)	Standard Deviation (%)	Relative Standard Deviation (%)	Reference Page(s)
Low QC 15.0µg/mL	1	14.0	15.0	93.3	104	6.90	6.63	V7
	2	14.6	15.0	97.3				
Medium QC 30.0µg/mL	1	32.2	30.0	107				
	2	33.2	30.0	111				
High QC 60.0µg/mL	1	65.2	60.0	109				
	2	62.3	60.0	104				
Standards								
0.200µg/mL 6-0432-1G	1	0.202	0.200	101	100	9.1	9.10	V7
0.200µg/mL 6-0432-1G	2	0.166	0.200	83.0				
1.00µg/mL 6-0432-1F	2	1.10	1.00	110				
5.00µg/mL 6-0432-1E	1	5.42	5.00	108				
20.0µg/mL 6-0432-1D	1	17.7	20.0	88.5				
20.0µg/mL 6-0432-1D	2	21.6	20.0	108				
50.0µg/mL 6-0432-1C	1	51.5	50.0	103				
80.0µg/mL 6-0432-1B	1	78.6	80.0	98.3				
100µg/mL 6-0432-1A	1	100	100	100				

InterIntra QC and stdsexh.xls, ACN\_ATD\_6\_0  
10:11 PM 9/20/01

Analyst: \_\_\_\_\_

Reviewed by: \_\_\_\_\_

PM3006635915

## Validation of a Method for the Determination of Acetonitrile in Human Exhalate Using Thermal Desorption GC-NPD

Sample Identification	Replicate	Concentration (µg/mL)	Concentration Added (µg/mL)	Percent Recovery (%)	Mean (%)	Standard Deviation (%)	Relative Standard Deviation (%)	Reference Page(s)
LLOQ QC 7.50µg/mL	1	5.92	7.50	78.9	93.0	12.25	13.2	V8
	2	5.43	7.50	72.4				
	3	6.23	7.50	83.1				
	4	6.31	7.50	84.1				
	5	6.11	7.50	81.5				
	6	6.41	7.50	85.5				
Low QC 15.0µg/mL	1	14.9	15.0	99.3				
	2	14.4	15.0	96.0				
Low QC 15.0µg/mL(inj)	1	13.4	15.0	89.3				
	2	13.5	15.0	90.0				
Low QC 15.0µg/mL(F/T)	1	14.2	15.0	94.7				
	2	15.6	15.0	104				
	3	10.4	15.0	69.3				
	4	12.6	15.0	84.0				
	5	13.8	15.0	92.0				
	6	12.1	15.0	80.7				
Low QC 15.0µg/mL(REF)	1	17.3	15.0	115				
	2	16.7	15.0	111				
	3	17.5	15.0	117				
	4	14.8	15.0	98.7				
	5	13.8	15.0	92.0				
	6	17.1	15.0	114				
Medium QC 30.0µg/mL	1	27.4	30.0	91.3				
	2	29.6	30.0	98.7				
High QC 60.0µg/mL	1	56.7	60.0	94.5				
	2	58.2	60.0	97.0				
Standards								
0.200µg/mL 6-0432-1G	1	0.184	0.200	92.0	100	8.1	8.10	V8
0.200µg/mL 6-0432-1G	2	0.184	0.200	92.0				
1.00µg/mL 6-0432-1F	1	1.11	1.00	111				
1.00µg/mL 6-0432-1F	2	1.15	1.00	115				
5.00µg/mL 6-0432-1E	1	4.93	5.00	98.6				
20.0µg/mL 6-0432-1D	1	17.9	20.0	89.5				
20.0µg/mL 6-0432-1D	2	20.4	20.0	102				
50.0µg/mL 6-0432-1C	1	49.6	50.0	99.2				
80.0µg/mL 6-0432-1B	1	79.8	80.0	99.8				
100µg/mL 6-0432-1A	1	102	100	102				

InterIntra QC and stdsexh.xls, ACN\_ATD\_7\_0  
10:11 PM 9/20/01

Analyst: \_\_\_\_\_

Reviewed by: \_\_\_\_\_

PM3006635916

## Validation of a Method for the Determination of Acetonitrile in Human Exhalate Using Thermal Desorption GC-NPD

Sample Identification	Replicate	Concentration (µg/mL)	Concentration Added (µg/mL)	Percent Recovery (%)	Mean (%)	Standard Deviation (%)	Relative Standard Deviation (%)	Reference Page(s)
Low QC 15.0µg/mL	1	17.7	15.0	118	102	8.9	8.73	V9
	2	18.1	15.0	121				
Low QC 15.0µg/mL(RT)	1	15.4	15.0	103				
	2	14.1	15.0	94				
	3	15.4	15.0	103				
	4	15.7	15.0	105				
	5	14.9	15.0	99.3				
	6	16.0	15.0	107				
Medium QC 30.0µg/mL	1	34.7	30.0	116				
	2	33.3	30.0	111				
High QC 60.0µg/mL(inj)	1	59.1	60.0	98.5				
	2	57.5	60.0	95.8				
High QC 60.0µg/mL(F/T)	1	61.5	60.0	103				
	2	63.8	60.0	106				
	3	56.3	60.0	93.8				
	4	62.7	60.0	105				
	5	62.8	60.0	105				
	6	62.1	60.0	104				
High QC 60.0µg/mL	1	60.9	60.0	102				
	2	62.7	60.0	105				
High QC 60.0µg/mL(RT)	1	48.1	60.0	80.2				
	2	55.4	60.0	92.3				
	3	52.0	60.0	86.7				
	4	58.2	60.0	97.0				
	5	59.9	60.0	99.8				
	6	62.7	60.0	105				
Standards								
0.200µg/mL 6-0432-1G	2	0.201	0.200	101	100	7.7	7.70	V9
1.00µg/mL 6-0432-1F	2	1.10	1.00	110				
20.0µg/mL 6-0432-1D	2	17.3	20.0	86.5				
50.0µg/mL 6-0432-1C	1	51.1	50.0	102				
80.0µg/mL 6-0432-1B	1	78.9	80.0	98.6				
100µg/mL 6-0432-1A	1	103	100	103				

InterIntra QC and stdsexh.xls, ACN\_ATD\_8\_0  
10:11 PM 9/20/01

Analyst: \_\_\_\_\_

Reviewed by: \_\_\_\_\_

PM3006635917

## Validation of a Method for the Determination of Acetonitrile in Human Exhalate Using Thermal Desorption GC-NPD

Sample Identification	Replicate	Concentration (µg/mL)	Concentration Added (µg/mL)	Percent Recovery (%)	Mean (%)	Standard Deviation (%)	Relative Standard Deviation (%)	Reference Page(s)
Low QC 15.0µg/mL	1	14.1	15.0	94.0	89.4	6.41	7.17	V11
	2	13.0	15.0	86.7				
	3	14.0	15.0	93.3				
	4	13.1	15.0	87.3				
	5	14.3	15.0	95.3				
	6	14.7	15.0	98.0				
Medium QC 30.0µg/mL	1	21.8	30.0	72.7				
	2	26.1	30.0	87.0				
	3	25.7	30.0	85.7				
	4	25.6	30.0	85.3				
	5	27.7	30.0	92.3				
	6	29.0	30.0	96.7				
High QC 60.0µg/mL	1	51.1	60.0	85.2				
	2	51.0	60.0	85.0				
	3	50.0	60.0	83.3				
	4	53.5	60.0	89.2				
	5	56.3	60.0	93.8				
	6	58.7	60.0	97.8				
Standards								
0.200µg/mL 6-0432-1G	1	0.181	0.200	90.5	100	7.5	7.52	V11
5.00µg/mL 6-0432-1E	1	5.67	5.00	113				
20.0µg/mL 6-0432-1D	2	19.1	20.0	95.5				
50.0µg/mL 6-0432-1C	1	50.7	50.0	101				
80.0µg/mL 6-0432-1B	1	79.1	80.0	98.9				
100µg/mL 6-0432-1A	1	100	100	100				

## Validation of a Method for the Determination of Acetonitrile in Human Exhalate Using Thermal Desorption GC-NPD

Sample Identification	Replicate	Concentration (µg/mL)	Concentration Added (µg/mL)	Percent Recovery (%)	Mean (%)	Standard Deviation (%)	Relative Standard Deviation (%)	Reference Page(s)
Low QC 15.0µg/mL	1	13.8	15.0	92.0	91.0	5.74	6.31	V12
	2	13.1	15.0	87.3				
	3	14.2	15.0	94.7				
	4	13.7	15.0	91.3				
	5	14.6	15.0	97.3				
	6	15.1	15.0	101				
Medium QC 30.0µg/mL	1	23.6	30.0	78.7	27.0	2.21	8.19	low conc SEE med conc SEE high conc SEE
	2	27.1	30.0	90.3				
	3	26.2	30.0	87.3				
	4	26.5	30.0	88.3				
	5	30.1	30.0	100				
	6	28.5	30.0	95.0				
High QC 60.0µg/mL	1	51.1	60.0	85.2	53.5	0.290	5.08	
	2	51.5	60.0	85.8				
	3	50.6	60.0	84.3				
	4	55.8	60.0	93.0				
	5	55.5	60.0	92.5				
	6	56.6	60.0	94.3				

a SEE = STD Deviation / SQRT (# of determinations).

SEE=STANDARD ERROR OF ESTIMATE

Standards								
0.200µg/mL 6-0432-1G	1	0.203	0.200	102	100	8.6	8.60	V12
0.200µg/mL 6-0432-1G	2	0.171	0.200	85.5				
5.00µg/mL 6-0432-1E	1	5.49	5.00	110				
20.0µg/mL 6-0432-1D	1	22.4	20.0	112				
20.0µg/mL 6-0432-1D	2	18.5	20.0	92.5				
50.0µg/mL 6-0432-1C	1	50.6	50.0	101				
80.0µg/mL 6-0432-1B	1	78.4	80.0	98.0				
100µg/mL 6-0432-1A	1	99.7	100	99.7				

## Validation of a Method for the Determination of Acetonitrile in Human Exhalate Using Thermal Desorption GC-NPD

Sample Identification	Replicate	Percent Recovery (%)	Mean (%)	Standard Deviation (%)	Relative Standard Deviation (%)	Reference Page(s)
Low QC 15.0µg/mL	1	99.3	91.0	6.06	6.66	ACN_ATD_7_0
	2	96.0				
Medium QC 30.0µg/mL	1	91.3				
	2	98.7				
High QC 60.0µg/mL	1	94.5				
	2	97.0				
Low QC 15.0µg/mL	1	94.0				ACN_ATD_9_0
	2	86.7				
	3	93.3				
	4	87.3				
	5	95.3				
	6	98.0				
Medium QC 30.0µg/mL	1	72.7				
	2	87.0				
	3	85.7				
	4	85.3				
	5	92.3				
	6	96.7				
High QC 60.0µg/mL	1	85.2				
	2	85.0				
	3	83.3				
	4	89.2				
	5	93.8				
	6	97.8				
Low QC 15.0µg/mL	1	92.0				ACN_ATD_10_0
	2	87.3				
	3	94.7				
	4	91.3				
	5	97.3				
	6	101				
Medium QC 30.0µg/mL	1	78.7				
	2	90.3				
	3	87.3				
	4	88.3				
	5	100				
	6	95.0				
High QC 60.0µg/mL	1	85.2				
	2	85.8				
	3	84.3				
	4	93.0				
	5	92.5				
	6	94.3				

InterIntra QC and stdsexh.xls, Inter-assay QC  
10:11 PM 9/20/01

Analyst: \_\_\_\_\_

Reviewed by: \_\_\_\_\_

PM3006635920

InterIntra QC and stdsexh.xls, Inter-assay QC  
10:11 PM 9/20/01

Analyst: \_\_\_\_\_

Reviewed by: \_\_\_\_\_

PM3006635921



## Validation of a Method for the Determination of Acetonitrile in Human Exhale Using Thermal Desorption GC-NPD

Sample Identification	Replicate	Percent Recovery (%)	Calculated Concentration ( $\mu\text{g/mL}$ ) <sup>a</sup>	Mean (%)	Standard Deviation (%)	Relative Standard Deviation (%)	Reference Page(s)
Low QC 15.0 $\mu\text{g/mL}$	1	99.3	14.9	93.8	4.51	4.81	ACN_ATD_7_0 SEE
	2	96.0	14.4		1.21		
Low QC 15.0 $\mu\text{g/mL}$	1	94.0	14.1	14.1	0.68 0.182	b 4.82	ACN_ATD_9_0 concentration(1n $\mu\text{g/mL}$ for M SEE
	2	86.7	13.0				
	3	93.3	14.0				
	4	87.3	13.1				
	5	95.3	14.3				
	6	98.0	14.7				
Low QC 15.0 $\mu\text{g/mL}$	1	92.0	13.8				ACN_ATD_10_0
	2	87.3	13.1				
	3	94.7	14.2				
	4	91.3	13.7				
	5	97.3	14.6				
	6	101	15.2				

a Concentration ( $\mu\text{g/mL}$ ) = (15.0 $\mu\text{g/mL}$  x Percent Recovery) / 100.

S.D. = Standard Deviation

b SEE = STD Deviation / SQRT (# of determinations).

SEE=STANDARD ERROR OF ESTIMATE

lean and S.D.)

InterIntra QC and stdsexh.xls, Inter-assay QC (Low)  
10:11 PM 9/20/01

Analyst: \_\_\_\_\_

Reviewed by: \_\_\_\_\_

PM3006635923

## Validation of a Method for the Determination of Acetonitrile in Human Exhalate Using Thermal Desorption GC-NPD

Sample Identification	Replicate	Percent Recovery (%)	Calculated Concentration ( $\mu\text{g/mL}$ ) <sup>a</sup>	Mean (%)	Standard Deviation (%)	Relative Standard Deviation (%)	Reference Page(s)
Medium QC 30.0 $\mu\text{g/mL}$	1	91.3	27.4	89.2	7.50	8.41	ACN_ATD_7_0 SEE
	2	98.7	29.6		2.00		
Medium QC 30.0 $\mu\text{g/mL}$	1	72.7	21.8	26.8	2.25 0.601	8.40	ACN_ATD_9_0 concentration(ln $\mu\text{g/mL}$ for M SEE
	2	87.0	26.1				
	3	85.7	25.7				
	4	85.3	25.6				
	5	92.3	27.7				
	6	96.7	29.0				
Medium QC 30.0 $\mu\text{g/mL}$	1	78.7	23.6				ACN_ATD_10_0
	2	90.3	27.1				
	3	87.3	26.2				
	4	88.3	26.5				
	5	100	30.0				
	6	95.0	28.5				

a Concentration ( $\mu\text{g/mL}$ ) = (30.0 $\mu\text{g/mL}$  x Percent Recovery) / 100.

S.D. = Standard Deviation

b SEE = STD Deviation / SQRT (# of determinations).

SEE=STANDARD ERROR OF ESTIMATE

lean and S.D.)

InterIntra QC and stdsexh.xls, Inter-assay QC (Med)  
10:11 PM 9/20/01

Analyst: \_\_\_\_\_

Reviewed by: \_\_\_\_\_

PM3006635925

## Validation of a Method for the Determination of Acetonitrile in Human Exhalate Using Thermal Desorption GC-NPD

Sample Identification	Replicate	Percent Recovery (%)	Calculated Concentration (µg/mL) <sup>a</sup>	Mean (%)	Standard Deviation (%)	Relative Standard Deviation (%)	Reference Page(s)
High QC 60.0µg/mL	1	94.5	56.7	90.1	5.15	5.72	ACN_ATD_7_0 SEE
	2	97.0	58.2		1.38		
High QC 60.0µg/mL	1	85.2	51.1	54.0	3.09	5.72	ACN_ATD_9_0 concentration(In µg/mL for M SEE
	2	85.0	51.0				
	3	83.3	50.0				
	4	89.2	53.5				
	5	93.8	56.3				
	6	97.8	58.7				
High QC 60.0µg/mL	1	85.2	51.1		0.826	b	ACN_ATD_10_0
	2	85.8	51.5				
	3	84.3	50.6				
	4	93.0	55.8				
	5	92.5	55.5				
	6	94.3	56.6				

a Concentration (µg/mL) = (60.0µg/mL x Percent Recovery) / 100.

S.D. = Standard Deviation

b SEE = STD Deviation / SQRT (# of determinations).

SEE=STANDARD ERROR OF ESTIMATE

lean and S.D.)

InterIntra QC and stdsexh.xls, Inter-assay QC (High)  
10:11 PM 9/20/01

Analyst: \_\_\_\_\_

Reviewed by: \_\_\_\_\_

PM3006635927

## Validation of a Method for the Determination of Acetonitrile in Human Exhalate Using Thermal Desorption GC-NPD

Sample Identification	Replicate	Concentration (µg/mL)	Mean (%)	Standard Deviation (%)	Relative Standard Deviation (%)	Reference Page(s)
Standards						
0.200µg/mL 6-0432-1G	1	92.0	100	7.8	7.8	ACN_ATD_7_0
0.200µg/mL 6-0432-1G	2	92.0				
1.00µg/mL 6-0432-1F	1	111				
1.00µg/mL 6-0432-1F	2	115				ACN_ATD_9_0
5.00µg/mL 6-0432-1E	1	98.6				
20.0µg/mL 6-0432-1D	1	89.5				
20.0µg/mL 6-0432-1D	2	102				ACN_ATD_10_0
50.0µg/mL 6-0432-1C	1	99.2				
80.0µg/mL 6-0432-1B	1	99.8				
100µg/mL 6-0432-1A	1	102				ACN_ATD_10_0
0.200µg/mL 6-0432-1G	1	90.5				
5.00µg/mL 6-0432-1E	1	113				
20.0µg/mL 6-0432-1D	2	95.5				ACN_ATD_10_0
50.0µg/mL 6-0432-1C	1	101				
80.0µg/mL 6-0432-1B	1	98.9				
100µg/mL 6-0432-1A	1	100				ACN_ATD_10_0
0.200µg/mL 6-0432-1G	1	102				
0.200µg/mL 6-0432-1G	2	85.5				
5.00µg/mL 6-0432-1E	1	110				ACN_ATD_10_0
20.0µg/mL 6-0432-1D	1	112				
20.0µg/mL 6-0432-1D	2	92.5				
50.0µg/mL 6-0432-1C	1	101				ACN_ATD_10_0
80.0µg/mL 6-0432-1B	1	98.0				
100µg/mL 6-0432-1A	1	100				

InterIntra QC and stdsexh.xls, Inter-assay Stds  
10:11 PM 9/20/01

Analyst: \_\_\_\_\_

Reviewed by: \_\_\_\_\_

PM3006635928